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# The UPDATE

December 2008

#### What's New

There have been several changes with our program since the last UPDATE. We have a new laboratory certification officer, Mahtab Gowan, who moved to our program in January 2008 from our state laboratory. At the beginning of September the Office of Environmental Laboratory Certification moved from Building #9 State Park, SC to the EQC Administration Building at 101 Business Park Blvd. Columbia, SC. While we are not far from our old office, we do have new shipping and mailing addresses. Please note the new addresses below:

New Mailing Address
Office of Environmental Laboratory Certification
SC DHEC
2600 Bull St.
Columbia, SC 29201

New Shipping Address
Office of Environmental Laboratory Certification
SC DHEC
101 Business Park Blvd.
77 Business Center
Columbia, SC 29203

Please note that the new shipping address must be used in order for packages to reach us. Some shipping services may charge an additional fee if the incorrect address is used. The phone and fax numbers have remained the same, so if you have any questions do not hesitate to call us. Please make the appropriate changes to update your records and come by to see us in our new offices if you have a chance.

## Proposed Fee Increase

On September 11, 2008, the DHEC Board granted staff initial approval to public notice the proposed amendments of R.61-30 to increase the certification fees. A Notice of Proposed Regulation, containing the text of the amendments and notice of opportunity for public comment, was published in the S.C. State Register on September 26, 2008, and can be viewed on the S.C. Legislature Online website at <a href="http://www.scstatehouse.net/regs/4015.doc">http://www.scstatehouse.net/regs/4015.doc</a>. For additional information, contact Ms. Carol Smith, Phone: 803-896-0992; Email: <a href="mailto:smithcf@dhec.sc.gov">smithcf@dhec.sc.gov</a>. A link to the proposed regulation can also be located on the Office of Laboratory Certification website at <a href="https://www.scdhec.gov/labcert">www.scdhec.gov/labcert</a>.

### **General Reporting Requirements**

General reporting requirements for analytical data submitted for regulatory compliance are specified by the Program area and/or project specifications. The Office of Environmental Laboratory Certification policy, unless otherwise stated by the Program area or for a specific project, is to report results to meet practical quantitation limits (PQLs) or reporting limits. This Office considers the PQL as the laboratory's established reporting limit. Certain Environmental Protection Agency (EPA) approved methodologies document PQLs or reporting limits in the methods themselves. The PQLs or reporting limits are determined using the lowest "non-zero" standard of the analyte included in the initial calibration curve and factoring in sample volume/weight used in the preparation step, final extract volume, along with other dilution factors. Analytical results reported below the laboratory's established PQL or reporting limit are not acceptable for regulatory compliance determinations with the exception of those documented at 40 CFR Part 141 for the Safe Drinking Water Act. Refer to 40 CFR Part 141 for reporting criteria for drinking water regulatory analyses.

With the exception of the Safe Drinking Water Act, analytical results must not be reported using the laboratory's method detection limits since these are established according to a statistical calculation at 40 CFR Part 136, Appendix B. These determinations, in certain instances, do not represent valid numbers for making regulatory decisions.

#### Volatiles

For example, for the volatile analyte benzene analyzed using EPA Method 624, the lowest concentration of benzene used for initial calibration is 1.0 µg/L. The PQL or reporting limit for benzene using EPA Method 624 is 1.0 µg/L.

#### Semivolatiles

For example, for the semivolatile analyte chrysene analyzed using EPA Method 625, the lowest concentration of chrysene used for initial calibration is 10 mg/L. 1000 milliliters of sample is extracted and concentrated to a final volume of 1.0 milliliter. Based on these factors with no dilutions performed on the extract, the PQL or reporting limit for chrysene using EPA Method 625 is  $10\mu g/L$ .

#### <u>Inorganics</u>

For example, for the inorganic analyte sulfate using ion chromatography or spectrophotometric analysis, use the lowest concentration of the analyte included in the initial calibration curve factoring in any dilution factor for the sample. If the lowest standard used for sulfate analysis is 5 mg/L, then the PQL or reporting limit for sulfate is 5 mg/L.

If reporting analytical results near the PQL or reporting limit, it is important to verify the accuracy of results at this concentration. Daily calibration verification is normally verified at the mid range of the calibration curve, therefore accuracy at the PQL or reporting limit must always be determined to verify the presence or absence of an analyte at this concentration unless otherwise stated in the method.

As noted above, SC DHEC considers data reporting to be a project-specific issue. Therefore, data reporting forms and qualifiers are not specified in this section. If the laboratory results reported on a Certificate of Analysis do not include the definitions of the qualifiers being used, then contact the laboratory and request that information. Then contact the applicable SC DHEC representative to determine if the qualified data is acceptable for regulatory compliance.

#### **NDPES Practical Quantitation Limits**

An updated Table of Practical Quantitation Limits (PQL) for NPDES sample compliance reporting for wastewater can be located on our website at <a href="https://www.scdhec.gov/labcert">www.scdhec.gov/labcert</a> under "Reporting Requirements." This table was recently revised to incorporate changes required under the Methods Update Rule which removed many EPA Methods as acceptable methods for analysis. It is important to note the footnotes at the bottom of the table which include specific requirements for method reporting. For parameters with both an EPA approved method and "Other Method" listed, the most stringent quality control criteria between the two methods must be met. This includes but is not limited to calibration criteria and quality control criteria for matrix and laboratory spikes. If there is not an approved NPDES EPA Method available for some of the analytes, the laboratory may use the SW-846 approved method in conjunction with the approved EPA NPDES Method. For instance, when reporting volatiles, some analytes are approved by EPA Method 624 whereas other analytes are only listed in SW-846 Method 8260B. When both methods are used, both methods must be referenced on the Certificate of Analysis and the most stringent quality control criteria between the methods must be met. It is not acceptable to only report SW-846 Method 8260B when some of the analytes are approved under EPA Method 624.

# **Certificate of Analysis**

Analytical results from the laboratory performing the analysis must be provided on a Certificate of Analysis. If analyses are contracted to other certified laboratories, then a Certificate of Analysis must be provided by each laboratory performing analyses. At a minimum, the following documentation must be provided on the Certificate of Analysis:

- 1) Laboratory Name
- 2) SC Laboratory ID Number
- 3) Sample Identification Number
- 4) Date and Time of Collection
- 5) Matrix
- 6) Sample Preparation Method (if applicable)
- 7) Date of Sample Preparation (if applicable)
- 8) Sample Analysis Method
- 9) Date of Sample Analysis
- 10) Analyte (s)
- 11) Analytical Result (at or above the specified PQL or reporting limit)
- 12) Laboratory PQL or Reporting Limit (for each analyte by method)
- 13) Dilution Factor (if employed)
- 14) Analyst
- 15) Qualifiers (List of definitions must be provided for any qualifiers used)
- 16) Contract Laboratory (Must be documented for each parameter and/or method)

The Certificate of Analysis is only valid when appearing on the Laboratory's Letterhead with the Laboratory Director's signature verifying the validity and accuracy of the analytical results. Specific quality control data should be requested as necessary to validate the analytical results.

## **Contract Laboratory Policy**

According to State Regulation 61-81, any laboratory which sub-contracts analytical work to another must establish that the contract laboratory has been certified by the Department for the appropriate method, parameter(s) and/or analyte(s). A copy of the current certificate for each contract laboratory should be requested and reviewed each time samples are sub-contracted, since a laboratory's certification is subject to change. The contract laboratory must also be informed of required PQLs or reporting limits along with the methodology required for the requested testing. Laboratory records along with a

Certificate of Analysis must indicate who performs the analyses and the name of the contract laboratory must be included in the records with their SC Laboratory ID number. A Certificate of Analysis must be provided by the contract laboratory meeting the requirements addressed above and be available for review. Questions concerning reporting requirements should be addressed to the Program area or project-specific contact. Questions concerning the certification status of a particular laboratory should be addressed to the Office of Environmental Laboratory Certification.

## SW-846 Notice of Availability of Final Update IV Certification Updates

On January 3, 2008, the EPA provided notice of the availability of "Final Update IV" to the Third Edition of the manual, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA publication SW-846. Final Update IV contains new and revised analytical methods that may be used in monitoring or complying with the Resource Conservation and Recovery Act (RCRA) hazardous waste regulations.

The South Carolina Certification Program is incorporating these new and revised methods into the Solid and Hazardous Waste Certification. The new and revised analytical methods can be found at: <a href="http://www.epa.gov/epaoswer/hazwaste/test/main.htm">http://www.epa.gov/epaoswer/hazwaste/test/main.htm</a>. Also be advised that some methods have been deleted. The deleted methods will be removed from the list of certified methods upon completion of this process.

Your laboratory should have already reviewed the methods you are certified to perform to determine if your present certification was affected by this rule. Standard Operating Procedures must have been updated to reflect the revised methods and new SOPs must have been developed for the new methods. The new or revised SOPs must have been submitted to our Office by **October 1, 2008** to enable us to review them and update your certification by December 31, 2008.

For out-of-state laboratories, updated certificates from your certifying authority reflecting certification for the new or revised methods and/or analytes must be submitted with the new or revised SOPs. If you encounter delays in receiving an updated certificate from your certifying authority, please contact our Office.

It is our goal to have all the certificates updated by December 31, 2008 to reflect the incorporation of the new and revised SW-846 methods. If you have any questions, please do not hesitate to contact us.

#### Spore Use for Sterilization Efficiency Verifications

The EPA requires the use of spore ampules and/or strips that contain live organisms (usually *Bacillus stearothermophilus*) to verify the sterilization efficiency of autoclaves and ovens used in laboratories performing microbiological analyses. This verification is required monthly. Spore ampules are used for autoclaves, while spore strips are used for hot air ovens. The basic process is to put one ampule or strip in with a load of items to be sterilized, while placing a second ampule or strip on the counter as a control. After the sterilization cycle is complete, remove the ampule or strip from the autoclave or oven, and then incubate it and the control according to the manufacturer's directions. After incubation, only the control should exhibit growth. A color change is used to indicate bacterial growth. Refer to the manufacturer's instructions for specific procedures. Results for the use of spore ampules or strips must be documented in the laboratory's sterilization records.

### Microbiological Wastes Must be Autoclaved

It is not acceptable to disinfect microbiological sample waste and contaminated equipment by adding chlorine bleach to the samples or by soaking the equipment in a bleach solution. Samples and contaminated equipment would have a high chlorine demand. While the addition of chlorine bleach would reduce the levels of microorganisms, it would not fully sterilize the samples. Also, the use of chlorine would produce chloramines. The intent is to reduce the level of chloramines in the environment because they are toxic to aquatic life and may be carcinogenic. In addition, it would be difficult to determine the contact time needed for each batch of contaminated materials since each batch would have a different chlorine demand. Furthermore, the practice of adding large amounts of chlorine bleach to the sewer system may violate state or local laws. The only technique acceptable to EPA is for all contaminated microbiological materials and samples to be sterilized by autoclaving for at least 30 minutes at 121°C. The sterilization of each batch of microbiological waste must be fully documented.

# A-1 Medium for Enumerating Fecal Coliforms in Ambient and Waste Water

The Methods Update Rule, (March 12, 2007) updated the holding time requirements for prepared A-1 medium. The Federal Register (40 CFR 141.74), footnote 8, now reads as follows: A–1 broth may be held up to 7 days in a tightly closed screw cap tube at 4 °C. Therefore, prepared A-1 tubes must be held no longer than 7 days. This includes commercially prepared A-1 medium tubes. This shorter hold time will likely preclude the use of commercially purchased prepared A-1 medium tubes as they cannot usually be received within 7 days of preparation. This hold time update applies to all laboratories that use A-1 medium for fecal coliform analyses.

## E. coli Certification for NPDES Water Testing

The EPA has approved *E. coli* testing as an alternative to fecal coliform testing for NPDES permits. However, South Carolina water quality standards (R.61-68, Water Classifications & Standards) mandate the use of fecal coliform testing. Therefore, use of *E. coli* methods are not approved at this time in South Carolina for NPDES analyses. If the water quality standards are changed, then NPDES permits may be changed accordingly. Testing is underway to determine typical ratios of *E. coli* vs. fecal coliforms in various water matrices across the State. Laboratories will be notified should *E. coli* testing become approved for NPDES analyses in South Carolina.

Certification for *E. coli* methods is available, however for laboratories that wish to analyze wastewater samples for *E.coli* for non-regulatory purposes such as process control, in-house comparisons, etc. Certification requirements will include the analysis of a WP Proficiency Testing (PT) sample for *E. coli*. Please contact this Office for more specific information.

# **Proficiency Testing Requirements**

The laboratory must participate in Water Supply (WS) and/or Water Pollution (WP) studies annually as required. The laboratory must obtain samples that are part of an official study obtained from an A2LA-approved provider. A list of approved PT providers may be found on the A2LA website at <a href="https://www.a2la.org">www.a2la.org</a>. A list of required PT parameters for South Carolina for WS and WP can be located on our website at <a href="https://www.scdhec.gov/labcert">www.scdhec.gov/labcert</a>.

In order to maintain certification in South Carolina, acceptable PT sample results for the laboratory must be received by December 31st each year for the Water Supply (WS) and/or Water Pollution (WP) studies. This means that the study the laboratory participates in must begin in the calendar year and end in the calendar year with the results received in our Office by December 31. The PT Provider must submit these

studies to this Office. Studies received in January will not be accepted for meeting the annual PT requirement. If acceptable PT Studies are not received by December 31st, decertification may be initiated.

All PT samples must be part of an official WS and/or WP study. Quick turn-around PT samples are not acceptable. Results must be sent to the provider prior to the study close date. No late results will be accepted. When completing the reporting forms to be sent to the PT Provider, you must indicate that the results are to be sent directly to this Office. The laboratory's EPA Lab Code and the State Lab ID number must also be included.

When completing the reporting forms, be sure to include the correct method number(s) being used. It is critical that the proper method number be referenced since PT samples are now required for each method for which you are certified. To ensure that you are reporting the correct method, review your certificate. If an incorrect method is reported, the PT result will not be considered acceptable. This includes reporting outdated methods that were removed by EPA in the Methods Update Rule published March 2007.

### **Proficiency Testing Sample Analysis Records**

Proficiency testing (PT) samples are to be treated in the same manner as regular samples being analyzed for compliance. Therefore, sample analysis records must be maintained for PT samples. During on-site evaluations, we will be reviewing PT sample analysis records for all PT samples. These records must be traceable to any quality control and calibration records as well as the proficiency testing final report from the PT provider. These PT sample analysis records must also document the analyst and the date and time of analysis. Please ensure that all PT sample analysis records are available for review for the on-site evaluation.

## New WS PT Requirement for Laboratories Certified for Compliance Monitoring

Beginning January 1, 2009, the new Fields of Proficiency Testing table for drinking water analytes which has been adopted by the NELAP Board becomes effective. Changes were made to establish acceptance limits for quantitative microbiological tests involving Membrane Filtration, Pour Plate, and Most Probable Number enumeration methods. As a result, laboratories that are certified for total coliform, fecal coliform, or *E. coli* enumeration methods are now required to successfully analyze a WS PT sample for enumeration to maintain certification. For example, if your laboratory is certified for SM 9223B using the Colilert MPN method for the enumeration of total coliform and *E. coli* then a PT sample must be reported for both. A number of approved PT providers already have this WS PT quantitative sample available. If your laboratory is certified for any total coliform, fecal coliform, or *E. coli* enumeration methods, please make arrangements to analyze this PT sample on an annual basis beginning in 2009. This PT requirement is in addition to the current requirement for analyzing a presence/absence PT for total coliform, fecal coliform, and *E. coli*. If you have any questions regarding this new WS PT requirement, please contact Alfred Baquiran at (803) 896-0977 or baquiraj@dhec.sc.gov.

#### WP Proficiency Testing Studies Are Now Required for SW-846 Methods

Our Office will begin requiring proficiency testing samples for SW-846 Methods beginning January 1, 2009. At this point, only the methods for water are required to be reported. PT samples for soils will not be required at this time, but will possibly be added in the future. The Water Pollution studies will be used for the SW-846 Methods. The table of required parameters is derived from The NELAC Institute's Fields of Proficiency Testing Table for Non-Potable Water. A list of parameters for which PTs are required is listed on our website at <a href="www.scdhec.gov/labcert">www.scdhec.gov/labcert</a>. For any questions regarding this certification change, please contact Susan Butts at (803) 896-0978 or <a href="mailto:buttsse@dhec.sc.gov">buttsse@dhec.sc.gov</a>.

### Method Flexibility from 40 CFR Part 136.6 for Clean Water Act Methods Only

40 CFR Part 136.6 addresses method modifications that are permitted for Clean Water Act methods. According to the EPA, this regulation allows these modifications to lower the cost of measurements, overcome matrix interferences, or to improve the analysis method without EPA review. The allowable changes to the methods are not to affect the chemistry of the method and the modified method must meet all quality control requirements from the original method to ensure equivalent performance. Please note that the method flexibility only applies to Clean Water Act methodology. This flexibility is not allowed under the Safe Drinking Water Act. In addition, a laboratory may not modify an approved analytical method for a method-defined analyte. This situation can cause conflict if a certain method is used to analyze both wastewater and drinking water samples. For example, EPA Method 245.1, Revision 3.0 (1994) is approved for mercury analysis under the Clean Water Act and Safe Drinking Water Act. Suppose a laboratory applies a modification to the method such as substituting stannous sulfate reagent for stannous chloride. While this modification is allowed under the method flexibility for Clean Water Act, it is not allowed for Safe Drinking Water Act. Laboratories have to be mindful of what types of samples are being analyzed using a specific method before implementing any method modifications. For any questions regarding method flexibility, please contact our Office at (803) 896-0970.

### **Oxygenate Certification**

We now offer certification for the oxygenate compounds by EPA Method 8260B. The Underground Storage Tank (UST) program will begin requiring all oxygenate data being reported to the Department to be analyzed by a certified laboratory by April 2009. Applications should be submitted as soon as possible in order to process and review the applications to meet this deadline. The oxygenate compounds will be certified separately from the other volatile compounds due to different QC requirements and due to the fact that most of the compounds are not included in Method 8260B which is the current approved method for volatiles. The certificates will read "EPA 8260B-OXY". The laboratory may apply for any approved volatile preparation methods, e.g. 5030B, 5035, etc. For a list of the oxygenate compounds and guidance on obtaining certification, please visit our website at <a href="https://www.scdhec.gov/labcert">www.scdhec.gov/labcert</a> for the oxygenate certification guidance document.

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